IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of
Inoue et al.
Serial No. 08/987,380
Filed: December 9, 1997
For: Granular Pesticidal Composition

Art Unit 1617
Examiner: WANG, SHENGJUN

DECLARATION

Honorable Commissioner of
Patents & Trademarks
Washington, D. C. 20231

Sir:

I, Masao INOUE, a citizen of Japan, residing at 298-1-503, Setogaya-cho, Hodogaya-ku, Yokohama-shi, Kanagawa, Japan, declares:

That I graduated from Kyoto University, Faculty of Engineering, Department of Polymer Chemistry in March 1988, and from the master course of said university in March 1990;

Then I entered Sumitomo Chemical Company, Limited in April 1990, and from then to June 2002, I had been engaged in studies of pesticidal formulation in Agricultural Chemicals Research Laboratory of said company and from July 2002, I have been engaged in management of formulation research and production in Planning & Coordination Office, Agricultural Chemicals Sector of said company.

That I conducted the following experiment to show effects of the invention of the above-identified application:

Experiment

<Production Example (1)>

Four parts by weight of N-(1,1,3-trimethyl-2-oxa-4-indanyl)-5-chloro-1,3-dimethylpyrazole-4-carboxamide [Furametpyr] and 0.8 parts by weight of hydrated silica were completely mixed in a juice mixer, and then, the mixture was ground by a pin mill. The resulting ground material has an average particle size of 19.1 μm (measured value by Coulter Counter TA II type).

The resulting ground material (4.8 parts by weight) obtained above, 20 parts by weight of Bentonite Fuji (bentonite manufactured by Hojun Yoko K.K.), 2 parts of Gohsenol GL-05 (manufactured by Nippon Synthetic Chemical Industry Co., Ltd.), 2 parts by weight of Sorpol 5060 (Toho Chemical Co., Ltd.) and 71.2 parts by weight of Fubasami clay A300 (clay manufactured by Fubasami Clay Industry Co., Ltd.) were completely mixed by a juice mixer, and the mixture was transferred to a mortar, and to this was added 20 parts by weight of water, and they were completely kneaded. The resulting kneaded material was granulated by a laboratory extruding granulator equipped with a 0.9 mmϕ die plate, and the particle size of the obtained granules was made uniform. Then, the granules was dried at 60°C for 15 minutes to obtain the pesticidal active ingredient-containing granule having a particle size from 1400 to 700 μm.

One thousand grams of the resulting pesticidal active ingredient-containing granule was rolled at 20 to 30 rpm in an inclined pan type rolling granulator which could control temperature equipped with a hot air generator, and to this was added 5 g of a mixture obtained by mixing 46.5 parts by weight of a polymeric MDI (mixture having a polymerization degree of 1 to 3), 52.5 parts by weight of trifunctional polypropylene glycol polyol and 1 part by weight of 2,4,6-tris(dimethylaminomethyl)phenol with maintaining the temperature at from 65 to 70°C, and the temperature of the mixture was kept at from 70 to 80°C for 3 minutes. The same operation that the mixture of the polymeric MDI, the trifunctional polypropylene glycol polyol and the

2,4,6-tris(dimethylaminomethyl)phenol was added and the resulting mixture was kept at from 70 to 80°C for 3 minutes was repeated 30 times, and finally, the resulting mixture was kept at from 65 to 70°C for 10 minutes to obtain a granular pesticidal composition (1) of the present invention.

The resultant obtained by adding the polymeric MDI, the trifunctional polypropylene glycol polyol and the 2,4,6-tris(dimethylaminomethyl)phenol to the pesticidal active ingredient-containing granule had good condition for mixing, although it had a middle viscosity. The granular pesticidal composition (1) was uniformly coated , and no agglomerated granules were observed.

<Comparative Production Example (1)>

The pesticidal active ingredient-containing granule was obtained using the same method as the Production Example (1).

One thousand grams of the pesticidal active ingredient-containing granule was rolled at 20 to 30 rpm in an inclined pan type rolling granulator and to this was added 100 g of a mixture of 90 g of the trifunctional polypropylene glycol polyol and 10 g of the 2,4,6-tris(dimethylaminomethyl)phenol. The resultant was mixed moreover to give granules containing the pesticidal active ingredient and the polyol.

One thousand and one hundred grams of the granules obtained was rolled at 20 to 30 rpm in an inclined pan type rolling granulator which could control temperature equipped with a hot air generator, to this was added 60 g of the polymeric MDI with maintaining the temperature at from 65 to 70°C. Then, the resultant was mixed at 65 to 70°C for 60 minutes to obtain a comparative composition (1).

The resultant obtained by adding the polymeric MDI to the granules containing the pesticidal active ingredient and the polyol had a remarkable viscosity and no good condition for mixing in the granulator. The comparative composition (1) contained not only coated granules but also a lot of agglomerated granules.

<Comparative Production Example (2)>

The granules containing th pesticidal active ingredient and the polyol was obtained using the same method as the Comparative Production Example (1).

One thousand and one hundred grams of the granules obtained was rolled at 20 to 30 rpm in an inclined pan type rolling granulator which could control temperature equipped with a hot air generator, to this was added 10 g of the polymeric MDI with maintaining the temperature at from 65 to 70°C. Then, the resultant was mixed at 65 to 70°C for 20 minutes. The same operation that the polymeric MDI was added and the resulting mixture was mixed at 65 to 70°C for 20 minutes was repeated 5 times, to obtain a comparative composition (2).

The comparative composition (2) contained not only coated granules but also agglomerated granules.

The resultant obtained by adding the polymeric MDI to the granules containing the pesticidal active ingredient and the polyol had a remarkable viscosity and no good condition for mixing in the granulator.

<Test Example>

Into a 500 ml beaker were charged the composition (1) and the comparative composition (2) obtained in the above Production Example 1 and Comparative Production Example 2 (300 mg, respectively) and 300 ml of 3° hardness water, and the mixture was stirred mildly. The temperature of the solution was kept at 25±1°C, and after given time, 1ml of the solution took out from the center part of the beaker was analyzed by gas chromatography to measure the amount of the pesticidal active ingredient therein, and release ratio was calculated appropriately by the following formula.

$$\text{Release ratio (\%)} = \frac{\text{Amount of pesticidal active ingredient (mg) in 1 ml of sample} \times 300}{\text{Initial amount of pesticidal active ingredient (mg) in 300 mg of composition tested}} \times 100$$

The results are shown in the following table.

	Release ratio (%)	
	14 days after	42 days after
Composition (1)	45	72
Comparative composition (2)	100	100

The undersigned declarant declares further that all statements made herein of his own knowledge are true an that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 31 day of October 2003.

Masao INOUE